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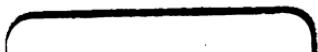
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**INORGANIC CHEMICAL  
PREPARATIONS**



# INORGANIC CHEMICAL PREPARATIONS

109-125

BY

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## P R E F A C E

THIS manual was written for the classes in Inorganic Preparations at the University of Chicago. The course is intended to familiarize advanced students with the methods and processes used in making inorganic compounds, and serves at the same time as a review in General Chemistry. It is supposed that the student who has finished the course is able without further assistance from an instructor to make any inorganic preparation. An attempt has been made to introduce all processes generally used, and at the same time to avoid repetition. The student should study each preparation in detail, work out other methods of making the same substance,

understand and criticise every step he takes and read some original articles. Therefore references to the literature have been given. No attempt has been made to make the list of references complete, as the student should learn to use dictionaries of chemistry, such as Dammer, Graham-Otto, etc.

The instructor must remember that the Manual is merely a laboratory guide, and that unless the work is carefully supervised it may become purely mechanical and the course lose half its value.

The preparations have not been arranged alphabetically or according to composition, but rather in the order of increasing difficulty. The methods have all been tried and have been found to give good results in the hands of careful students. A table of solubilities is appended.

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## GENERAL DIRECTIONS

A GAS should always be washed unless there is specific reason for not doing so. If the gas is very soluble in water, and washing would involve too much loss, it may frequently be purified by passing it into a litre bottle before using so that some of the impurities settle. If a dry gas is needed, the drying agent used depends upon the nature of the gas. Sulphuric acid, calcium chloride, calcium oxide, potassium hydroxide, etc., are commonly used, and the gas finally dried if necessary by phosphoric anhydride mixed with beads or by sodium. As the sodium must present a large surface, it may be used as wire or as the powder

obtained when it is melted under toluene and shaken violently (care). In generating a gas a safety tube should always be used when possible. When a gas is being absorbed by a liquid precautions should always be taken to prevent the liquid running back into the generating flask. For this purpose the delivery tube may dip under mercury, the wash-bottle may be provided with a valve, etc. An excellent device is to place between the wash-bottle and the absorption flask an empty reversed wash-bottle large enough to hold all the absorbing liquid. The long tube of the wash-bottle is next to the absorption flask, so that if liquid runs back it remains in the safety bottle as long as all the gas is absorbed, and any gas not absorbed forces it back into the absorption flask.

If a solid is formed by the action of a gas on a liquid, the delivery tube should

have a wide exit, and should dip just below the surface. A funnel tube is well adapted to this purpose.

Always absorb excess of chlorine, ammonia, sulphur dioxide, etc.

The gases generally used in the laboratory are hydrogen, oxygen, nitrogen, carbon dioxide, hydrochloric acid, chlorine, sulphur dioxide, and ammonia.

Hydrogen and carbon dioxide generators must always be on hand. Oxygen and nitrogen are most conveniently used from gasometers. Nitrogen may be made by heating gently a solution of 60 grammes each sodium nitrite, ammonium chloride, and potassium bichromate in 2 litres water. It is purified by passing over hot copper.

The most convenient source of gaseous hydrochloric acid is the concentrated solution. Some of this is placed in a flask with about one-sixth its weight of common salt

and concentrated sulphuric acid slowly added from a drop funnel. In a short time the gas is evolved, and may be controlled by regulating the flow of sulphuric acid. The drop funnel must have a long stem, and this must be full of sulphuric acid so as to overcome the pressure of the liquid in the wash-bottles.

A steady stream of chlorine may be obtained by heating in a water-bath one part pyrolusite and four parts crude hydrochloric acid contained in a flask or stone jug. The spent liquid should not be thrown away but worked for manganous chloride.

Sulphur dioxide is easily obtained by heating on asbestos board copper turnings with two or three times their weight of concentrated sulphuric acid. The addition of a few small pieces of charcoal is advisable. Unless entirely used up the acid should be separated from the powder that settles to the bottom and kept for future

use. The powder dissolved in water yields crystals of copper sulphate.

Gaseous ammonia is evolved when ammonia water is gently heated. It should be dried with lime and potassium or sodium hydroxide.

Liquids are usually purified by redistillation collecting the fraction or fractions with constant boiling-point. It may at times be necessary to distil under diminished pressure. For that purpose the apparatus of Brühl will be found convenient. Never put the distilling flask above the level of the eyes, and when distilling under diminished pressure always use goggles.

In distilling always leave an opening for the escape of any gas formed and of the air in the apparatus. The opening may be protected from the air and moisture by a mercury valve, a drying tube, etc. When a liquid cannot be distilled without decom-

position it may be identified by its color, density, index of refraction, etc.

Solids may usually be purified by recrystallization. If the substance is much less soluble in the cold than in the hot solvent, a hot saturated solution is first made and then cooled rapidly with constant stirring. After one or more repetitions the product is pure, and large crystals may now be obtained by dissolving in little cold solvent and allowing to evaporate slowly. Water is the usual inorganic solvent, but sometimes it is necessary to extract with alcohol, ether, etc. Occasionally the impure product is dissolved and the impurity or the substance desired thrown out by adding another liquid, in which it is insoluble.

Solids are sometimes purified by sublimation, though for obvious reasons this is less convenient and less used than the distillation of liquids.

Organic matter is frequently objectionable, and as corks and rubber are almost indispensable, they may be protected by wrapping thin asbestos paper around the stoppers or around the ends of the glass tubes connected by the rubber.

It is not usually necessary to use chemically pure reagents in making preparations. Common sense must here be the guide.

Complete notes should be kept. It is particularly important to record yield and purity of products.

## ABBREVIATIONS OF REFERENCES

A. Liebig's Annalen.  
A. ch. Annales de Chimie.  
A. ch. p. Annales de Chimie et de Physique.  
Am. J. Sc. American Journal of Science and Arts.  
A. Min. Annales des Mines.  
B. Berichte der deutschen chemischen Gesellschaft.  
Bl. Bulletin de la Société chimique de Paris.  
Ch. C. Chemisches Centralblatt.  
Ch. N. Chemical News.  
C. r. Comptes-rendus des Séances de l'Académie des Sciences.  
D. Dingler's Polytechnisches Journal.  
J. Jahresbericht über die Fortschritte der Chemie.  
J. pr. Journal für praktische Chemie.  
P. A. Poggendorf's Annalen.  
Ph. C. Pharmaceutische Centralhalle.  
Rec. Recueil des Travaux chimiques des Pays-Bas.  
Scher. Scherer's Journal der Chemie.  
Soc. Journal of the Chemical Society of London.  
Z. Zeitschrift für Chemie und Pharmacie.  
Z. Anorg. Zeitschrift für anorganische Chemie.  
Z. phys. Zeitschrift für physikalische Chemie.

## INORGANIC CHEMICAL PREPARATIONS

### SODIUM CHLORIDE

Wittstein, Buchner's Repertorium 65, 361.

Dissolve 150 grammes common salt in 500 c.c. water; filter, and to the hot solution add milk of lime containing 2½ grammes calcium hydroxide, then a slight excess of barium chloride. Allow to stand, filter, and add a solution of 5 grammes sodium carbonate; heat to boiling, filter, neutralize with dilute hydrochloric acid, and evaporate to 150 c.c. as rapidly as possible. Collect the sodium chloride on a platinum cone, wash with little hot water, and dry by

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heating gently in a platinum or porcelain dish.

### POTASSIUM CHLORATE

Liebig, A. 41, 307.

Slake 75 grammes quicklime, mix it with 30 grammes potassium chloride, and add sufficient water to make a thin paste. Heat almost to boiling, and pass in chlorine (using wide delivery tube) until no more is absorbed and all the lime has passed into solution. When necessary add water to keep the volume constant. Boil for one hour, passing carbon dioxide the last ten minutes, and filter hot. Evaporate to 100 c.c., and set aside to crystallize. Evaporate the mother liquor further, and again set aside. Purify the potassium chlorate by recrystallization until it is free from chloride.

## BICARBONATE OF SODA

Schlösing & Rollant, A. ch. p. (4) 14, 5.

Mond, Ch. C. 1886, 189.

Dissolve 100 grammes salt in 300 c.c. water, and saturate the solution with ammonia, keeping it cold with ice water. Filter if necessary, and put into a tall, narrow cylinder. Cork the cylinder, and have a wide glass tube pass through the cork, and dip about 20 centimetres below the surface of the liquid. Connect with a carbon dioxide generator (Kipp's), uncork the cylinder, and allow carbon dioxide to pass through. When all the air is expelled from the cylinder cork tightly, and allow carbon dioxide to pass in as rapidly and as long as it will, removing the cork from time to time. Filter from the bicarbonate, wash it with cold water, and spread it out to dry, covering it with filter paper. After

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two or three days again wash with ice water, and dry at 40°.

### SODIUM NITRITE

Hampe, A. 125, 336.

Melt 85 grammes sodium nitrate in a small, flat, iron dish (sand-bath), and add slowly 206 grammes granulated lead. Stir until the lead is completely oxidized (30-50 minutes), and pour the molten mass upon an iron or slate slab. Powder and wash thoroughly with boiling water. Cool the filtrate, pass in carbon dioxide for a few minutes, heat to boiling, and filter hot. Evaporate until crystals separate on cooling. Unless the evaporation has been carried too far, the crystals are almost pure sodium nitrate. Pour off the solution, again evaporate and cool, and repeat until the solid shows a decided test for nitrite. Then

evaporate the mother liquor to dryness, and extract repeatedly with boiling absolute alcohol. Evaporate the alcohol and recrystallize from water. To determine the strength of the nitrite, dissolve a weighed portion in considerable water, add the quantity of potassium permanganate it would require if pure, acidify with sulphuric acid, and titrate the excess of permanganate.

### BASIC LEAD NITRITE

Chevreul, A. ch. 83, 72.

Péligot, A. ch. p. (3) 2, 87.

v. Lorenz, Wien. Akad. Ber. (2) 84, 1133.

Dissolve 25 grammes lead nitrate in 2 litres water, add gradually 50 grammes finely granulated lead, and heat on the water-bath for several days. Cool in ice water, pass in carbon dioxide fifteen minutes, boil two or three minutes, filter hot, and evaporate to crystallization.

## CUPROUS CHLORIDE

Proust, Scher. 8, 49.

A. ch. 28, 218; 32, 48.

Dissolve 20 grammes cupric oxide in 200 c.c. commercial hydrochloric acid and heat with 25 grammes copper turnings until the green color disappears. Pour the liquid into a tall cylinder containing at least 1 litre distilled water. Allow the white precipitate to settle, and wash it with very dilute hydrochloric acid until the washings are colorless, decanting or siphoning off the liquid. Then bring the precipitate upon a filter, and, using a filter-pump, wash as rapidly as possible, first with very dilute hydrochloric acid, then with alcohol, and finally with ether. Until all the water is washed out, the precipitate should be kept covered with liquid so as to protect it from the air. If the precipitate is white after washing, bring it into a vacuum

desiccator over paraffine and sulphuric acid, and when dry keep in a tightly stoppered bottle. If the product is reddish or green, dissolve it in concentrated hydrochloric acid, pour into water, and wash as before.

## **LEAD DIOXIDE**

### **Dry Method**

*Kassner, D. 274, 136, 183, 226, 270.*

*Archiv der Pharmacie, 228, 109.*

Mix intimately 50 grammes each lead oxide and calcium carbonate, put into a clay roasting dish, and heat for thirty minutes to redness, stirring with a pipe-stem so as to expose the hot mass to the action of the air. Cool, powder, and again heat for thirty minutes, repeating the operation until the color no longer changes on heating. Add the powder gradually to 500 c.c. dilute nitric acid, decant, wash two or three times

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with 200 c.c. dilute nitric acid, then with hot water, and dry at 125°.

### **Wet Method**

Wöhler, A. 90, 383.

Rivot, Bendant and Daguin, A. Min. (5) 4, 221.

Böttger, J. pr. 76, 235.

Dissolve 50 grammes lead acetate in 200 c.c. water, add 200 c.c. sodium hydroxide solution (20%), and pass in chlorine as long as any action is observed. Decant, wash with water, boil with 300 c.c. dilute nitric acid, again wash with water, and dry at 125°.

### **MERCURIC OXIDE**

#### **Red Mercuric Oxide, Dry Method**

Heat cautiously 10 grammes mercuric nitrate in a porcelain crucible until red fumes cease to come off, and mercury condenses on a cold crucible cover held above the heated mass.

**Red Mercuric Oxide, Wet Method**

Bosetti, *Pharmaceut. Zeitung* 35, 471.

To a boiling solution of 30 grammes mercuric chloride in 100 grammes water add a hot solution of barium hydroxide 1:4, at first rapidly, then drop by drop, until the dark brown precipitate changes to deep red. Then pour into 4 litres hot water, wash, and dry.

**Yellow Mercuric Oxide**

Siewert, A. 125, 226.

Oswald, *Z. phys.* 18, 159.

Bosetti, *loc. cit.*

Dissolve 30 grammes mercuric chloride in 300 c.c. water, and pour the solution into 250 c.c. potassium hydroxide solution (10%). Wash and dry.

**BORIC ACID**

Dissolve 50 grammes borax in 200 c.c. boiling water, and add 30 c.c. concentrated

hydrochloric acid. Allow to stand 24 hours, and collect the boric acid on a filter. Wash it with little cold water, and heat to 150° for one hour; dissolve in little boiling water and allow to crystallize.

## BORON

Jones, Soc. 35, 41.

Gattermann, B. 22, 195.

Winkler, B. 23, 772.

Moissan, C. r. 114, 392.

Melt 250 grammes borax in a Hessian crucible, and when it reaches a state of quiet fusion pour upon a slab. Powder while hot, and mix at once 25 grammes with 12 grammes powdered magnesium. Put the mixture into the same crucible, pack tightly, and add a layer of the fused and powdered borax. Close the crucible with a well-fitting cover. (Work as rapidly as possible.) Heat for fifteen to twenty min-

utes to redness. When the crucible is cold break it and remove the contents, discarding carefully adhering pieces of crucible. Powder and boil successively with water, concentrated hydrochloric acid and water. The residue is impure boron. To purify, mix with 20 grammes fused borax in a Hessian crucible, add a layer of borax, cover the crucible, and put it in a bed of titanium oxide and charcoal in a larger crucible, surrounding it completely with the mixture. Heat and wash as before, repeating if necessary.

## SODIUM THIOSULPHATE

Walchner, A. 46, 235.

Divide into two equal portions a hot saturated aqueous solution of 200 grammes sodium carbonate. Keep one portion warm on the water-bath, pass through sulphur

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dioxide as long as it is absorbed, and add at once the other portion. Boil the solution of sodium sulphite with flowers of sulphur until no more dissolves. Filter, evaporate to a small bulk, and allow to crystallize. Purify by recrystallization.

BARIUM DITHIONATE

Gay-Lussac & Welter, A. ch. p. 10, 313.

Spring & Bourgeois, Bl. (2) 46, 151.

Suspend 50 grammes precipitated manganese dioxide in water, put the flask in ice water, and lead in sulphur dioxide as long as anything goes into solution. Filter, heat to boiling, cool, and add barium hydroxide solution until ammonium sulphide added to a small portion no longer gives a flesh-colored precipitate. Filter, wash the precipitate with boiling water, add the washings to the main filtrate, heat to boiling, and pass in carbon dioxide until the liquid is

neutral to litmus. Filter hot, evaporate, and allow to crystallize.

### **AMMONIUM NITRATE**

To 250 c.c. nitric acid kept cold by a freezing mixture, add slowly and with constant stirring concentrated ammonia water in slight excess. Allow to stand over night, and separate by decantation and filtration from any brown sediment. Should crystals separate, redissolve in little cold water and add to the main liquid. Evaporate on the water-bath until the density of the hot liquid is 1.25. Allow to crystallize, separate the crystals, dry them between filter paper, and work the mother liquor further.

### **SODIUM AMMONIUM PHOSPHATE**

Mitscherlich, A. ch. p. 19, 399.

Dissolve 375 grammes crystallized sodium phosphate in 125 c.c. water, and to the hot

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solution add slowly 65 grammes ammonium chloride. When all is dissolved boil for a moment and filter, using hot water funnel. Cool rapidly with constant stirring and drain the crystals. Redissolve in 75 c.c. boiling water and recrystallize adding a little ammonia. Repeat until free from sodium chloride. Dry at 30° and keep in well-closed bottle.

### ZINC—FREE FROM ARSENIC

L'Hôte, C. r. 98, 1491.

Melt 500 grammes zinc in a Hessian crucible, and by means of a thick iron wire hold 6 to 8 grammes magnesium chloride below the surface of the molten mass, stirring gently. When all the magnesium chloride has disappeared, heat almost to the boiling-point of zinc 15 minutes, then allow the zinc to cool somewhat and granulate by pouring into water.

## ARSENIC ACID

Kopp, C. r. 42, 1060.

In a retort heat 100 grammes arsenious oxide in small pieces (why not in powder?) with 100 c.c. fuming nitric acid. When fumes are no longer evolved, pour off the liquid and evaporate to dryness. Test for arsenious acid, and if any is present add aqua regia and again evaporate to dryness. Again test for arsenious acid and repeat evaporation with aqua regia until free from arsenious acid. Then dissolve in boiling water and evaporate to a thin syrup. Heat on the water-bath until crystals form on cooling.

## CHROME ALUM

Böttger, J. pr. 36, 318.

Traube, A. 66, 169.

Dissolve 100 grammes potassium bichromate in 400 c.c. warm water, add 140

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grammes sulphuric acid and cool to 30°. Add slowly alcohol until there is no further action, taking care to keep the temperature below 50°. Cool to zero and decant from the crystals. Evaporate the liquid to 100 c.c., keeping the temperature below 50°; again cool to zero and decant from the crystals. Combine the two crops and purify by recrystallization, keeping the temperature of the solution below 50°.

## CHROMIC ANHYDRIDE

Bolley, A. 56, 113.

Traube, A. 66, 169.

Dissolve 300 grammes potassium bichromate in 500 c.c. water, add 420 c.c. sulphuric acid, and allow to stand over night. Filter through glass wool and wash the acid potassium sulphate with not over 10 c.c. cold water. To the filtrate add 20 c.c. sulphuric

acid and evaporate until chromic acid begins to separate. Allow to stand over night and collect on a platinum cone. Wash with 50 c.c. nitric acid and dry in desiccator over potassium hydroxide.

### CHROMOUS ACETATE

Moissan, A. ch. p. (5) 25, 416.

Heat 50 grammes chromic anhydride with 500 c.c. fuming hydrochloric acid until no more chlorine is evolved. Evaporate to 200 c.c., pour the liquid upon 250 grammes granulated zinc in a large flask, and add 125 c.c. hydrochloric acid. When the first violent action is over, place in the neck of the flask a rubber stopper carrying two tubes, one terminating just below the stopper, the other going to the bottom of the flask. Connect the longer with a bent tube reaching below the surface of a solution of 400 grammes crystallized sodium acetate

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in  $1\frac{1}{2}$  litres water. When the liquid is a pure blue, close the shorter tube and the hydrogen evolved will force the liquid into the sodium acetate solution. If necessary filter the chromous chloride solution by inserting a bulb tube with glass wool. Wash the red precipitate of chromous acetate repeatedly with boiled water through which carbon dioxide has been passed, and keep as a paste. Keep the flask containing the chromous acetate full of carbon dioxide while washing, and siphon off the water used for washing.

## HYDROBROMIC ACID

Linnemann, A. 161, 198.

Mix 10 grammes red phosphorus with 80 grammes sand, put into a small flask and moisten with 20 grammes water. Close the flask with a stopper carrying a glass tube

and a drop funnel, and by means of the latter gradually drop in 60 grammes bromine. Pass the gaseous hydrobromic acid through a U-tube containing moist red phosphorus mixed with plenty of beads or pieces of glass and then into 60 grammes water. To prevent water running back, pass the delivery tube into the tubular of a small retort whose neck dips into the 60 grammes water contained in a beaker. Loss of bromine may be avoided and a steady flow obtained by closing the drop funnel with a cork through which a glass tube drawn to a capillary is passed. Determine the strength of the aqueous acid obtained.

### HYDRIODIC ACID

Naumann, A. 151, 145.

Berthelot, C. r. 76, 741.

Powder 50 grammes iodine, suspend it in 250 c.c. water in a 500 c.c. flask and pass

in hydrogen sulphide. Provide the flask containing the iodine and water with a stopper through which the delivery tube passes, and as soon as all the air is displaced, close the flask and leave it connected with the hydrogen sulphide generator over night. If the iodine has not all disappeared, lift the stopper every hour for a minute or two and shake vigorously from time to time. As soon as the iodine has disappeared, heat gently on water-bath passing hydrogen through to drive out excess of hydrogen sulphide. Filter from sulphur, and distil in a current of hydrogen, collecting the portion between  $125^{\circ}$  and  $130^{\circ}$ . Sometimes the iodine gets coated with sulphur and is not further acted upon. It must then be removed and powdered. If large quantities of hydriodic acid are to be made, it is advisable to dissolve the iodine instead of suspending it.

Fifty to sixty grammes hydriodic acid may be made as above and without first concentrating used to dissolve the iodine.

## TIN TETRACHLORIDE

Lorenz, Z. Anorg. 10, 44.

Seal one end of a glass tube 2 or 3 centimetres in diameter and 40 centimetres long and fit into it a double bored rubber stopper. Through one of the holes pass an inverted condenser, and through the other a long glass tube that may be raised or lowered. Fill the larger tube with granulated tin, have the delivery tube reach to the bottom and pass in dry chlorine. Liquid tin tetrachloride soon forms. Move the delivery tube so that it just dips into the liquid, and pass the chlorine until action ceases. Pour from the little tin usually left, purify by

redistillation and keep in sealed tubes. Tin tetrachloride boils at 114°.

## SULPHURYL CHLORIDE

Schulze, J. pr. (2) 24, 168.

Put 30 grammes camphor into an absorption flask, place in ice water and lead in sulphur dioxide as long as it is absorbed. Then lead in chlorine until it passes through unabsorbed, then sulphur dioxide and chlorine in rotation until the liquid no longer increases in bulk. Put into a retort and collect the portion coming over below 100°. If required, the portion boiling above 100° may be treated in the same way as the camphor, *i.e.* sulphur dioxide and chlorine led in alternately, and thus a large quantity of low boiling liquid obtained from little camphor. The low boiling liquid is sub-

jected to fractional distillation, the sulphuryl chloride coming over between 68° and 71°.

## PHOSPHORUS TRICHLORIDE

Michaelis, Graham-Otto's *Lehrbuch* II. 324.

Cover the bottom of a tubulated retort with dry sand and connect with a double-necked receiver, protecting the corks with asbestos paper. In the second neck of the receiver put a drying tube and absorb the gas which passes this in sodium hydroxide solution, using a safety bottle or mercury valve. (If no double-necked receiver is at hand, an ordinary flask and double-bored cork may be used.) Fill the apparatus with carbon dioxide, throw in 100 grammes yellow phosphorus dried between filter paper, and then dipped successively into alcohol and ether, again pass carbon dioxide for fifteen minutes, put the retort into a pail of warm

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water and pass in a current of chlorine. The chlorine delivery tube must move easily in the stopper so that it may be raised or lowered. If the end of the tube is too near the phosphorus, the latter gets hot and distils, forming a red crust. If the tube is too far away, the action is slow, the excess of chlorine acts and phosphorus pentachloride is formed. The position must be such that the retort remains uncoated. As soon as action begins, as indicated by a tongue of flame from the tube to the phosphorus, the retort may be removed from the hot water, but water should always be kept under the retort to catch the phosphorus in case of accident. Toward the end, the voluminous phosphorus remaining may be cautiously heated. Purify the trichloride by redistillation, excluding water. Be careful not to inhale any. Boiling-point 74°.

## FLUORSILICIC ACID

Berzelius, J. 1825, 89; P. A. 1, 169.

Stolba, D. 197, 336.

Mix 50 grammes each fluor-spar and sand, and heat in an old flask with 150 c.c. sulphuric acid. Lead the gas evolved through an empty flask, and then into 200 c.c. water, using a mercury valve or a thistle tube. When the action is over press the liquid from the separated silicic acid, wash with little water, and filter. Determine strength of acid by titration.

## ANTIMONY TRICHLORIDE

In a 500 c.c. flask heat 50 grammes gray antimony ore with 250 c.c. concentrated hydrochloric acid, adding gradually a few grammes potassium chlorate. As soon as all the ore is acted upon filter through

glass wool and evaporate to 100 c.c. Pour into 2000 c.c. water, wash the precipitate with cold water, and dissolve it in 75 c.c. concentrated hydrochloric acid, leading in gaseous hydrochloric acid until no more is absorbed. Pour the solution into a tubulated retort containing some bits of glass to prevent bumping, and distil. As soon as a drop of the distillate gives a white precipitate with water change the receiver, and do this again when the distillate begins to solidify on a glass rod. The last portion is antimony trichloride, a white crystalline substance. If colored by iron it must be redissolved in concentrated hydrochloric acid and redistilled. The portion of the distillate that gives a white precipitate with water is a solution of antimony trichloride in hydrochloric acid, and may be used for the next preparation.

**ANTIMONY OXYCHLORIDE**

Johnston, J. pr. 6, 55.

Malaguti, A. ch. p. 59, 220.

Péligot, A. ch. p. (3) 20, 288.

**Powder of Algaroth**

Pour the acid solution of antimony trichloride obtained in the previous experiment into 400–500 c.c. water, wash the precipitate carefully, dry and preserve.

**SILVER NITRATE**

Place a porcelain crucible in a Hessian crucible with a layer of sand between them. Cover the sand with fused borax, and heat to redness. Gradually bring into the hot porcelain crucible a mixture of 100 grammes silver chloride, 100 grammes dry sodium carbonate, and 15 grammes potassium nitrate. Add slowly, or it may foam over.

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When the mass is in quiet fusion stir with a pipe-stem, allow to settle, cool slightly, and pour the contents of the porcelain crucible into water. The coating of borax should keep sand from dropping in. Brush the silver clean, and boil for a few minutes with dilute sulphuric acid. Wash, dissolve in nitric acid, evaporate to dryness, and recrystallize from water.

From the silver residues of the laboratory (chloride, bromide, cyanide, etc.), the chloride may be obtained as follows: Dissolve the residues in 10% potassium cyanide solution, and digest on the water-bath with zinc dust until all silver is precipitated. Wash with water, boil with hydrochloric acid, again wash thoroughly with water, dissolve in nitric acid, and precipitate with hydrochloric acid. If necessary, redissolve in ammonia, and precipitate with hydrochloric acid.

## SILVER HYPONITRITE

Divers, Ch. N. 23, 206.

Divers & Haga, Soc. 45, 78.

Van der Plaats, B. 10, 1507.

Mühlhäuser, Z. 1864, 720.

Dissolve 10 grammes sodium nitrite in 30 grammes water, cool in ice water, and add slowly 250 grammes 3% sodium amalgam. When action ceases pour from the mercury, neutralize with acetic acid, and precipitate with silver nitrate. To get rid of any silver acetate in the precipitate expose to diffused sunlight for a few hours, dissolve in very dilute sulphuric acid, and precipitate with ammonia, carefully avoiding an excess. Wash with a little ice water, and dry in desiccator. Keep all solutions ice cold, and avoid direct sunlight.

To prepare sodium amalgam, heat sodium to 90° under low melting paraffine, and pour

### 30 INORGANIC CHEMICAL PREPARATIONS

in the mercury in a fine stream, stirring constantly. The operation is harmless, but should be carried on in the hood. When working with molten sodium use care, and protect the eyes with goggles.

### STRONTIUM CHLORIDE

Berthier, A. ch. p. 22, 231.

In a Hessian crucible heat to bright redness for two hours a mixture of 200 grammes celestine and 75 grammes charcoal. When cool powder, and add to 2 litres dilute hydrochloric acid 5 %. Heat to boiling, filter hot, and evaporate to dryness. Redissolve in water, remove iron, etc. by ammonium sulphide, and barium by digesting with strontium sulphate, and recrystallize.

### STRONTIUM HYDROXIDE

Dissolve 75 grammes strontium chloride in boiling water, add 75 grammes sodium

hydroxide solution (30 %), cool, collect on a platinum cone, and wash with cold water.

### MANGANOUS CHLORIDE

Everitt, Phil. Mag. 6, 193.

Kolbe, J. pr. (2) 5, 445.

Evaporate to dryness 1 litre of the liquid obtained in making chlorine from manganese dioxide and hydrochloric acid. Heat the residue for several hours, cool, wash with boiling water, and filter. To  $\frac{1}{10}$  of the filtrate add sodium carbonate, wash the precipitate, and digest it on the water-bath with the main portion until ammonium sulphide gives a fresh-colored precipitate entirely soluble in dilute acetic acid. Filter and recrystallize.

### MANGANESE

Glatzel, B. 22, 2857.

Heat 150 grammes manganous chloride until anhydrous, and mix 75 grammes of the

### 32 INORGANIC CHEMICAL PREPARATIONS

anhydrous salt with 100 grammes dry potassium chloride and 50 grammes anhydrous calcium chloride. Pack the mixture in a Hessian crucible, and heat to quiet fusion. Then add 18 grammes magnesium in pieces of 3 to 4 grammes. As each piece is thrown in cover the crucible, and wait until the action is over before adding another. If the mass is too hot it will foam over. Therefore, have it as cold as is consistent with complete fusion while adding the magnesium, but as soon as all the magnesium is in bring to a white heat as rapidly as possible. Keep at white heat 20-30 minutes. Allow to cool, break the crucible, and wash the regulus of manganese. If the mass was brought to a white heat immediately on adding the magnesium, the manganese will be in one or two pieces at or near the bottom. Otherwise it will be found as small, fairly stable pellets, or as an

unstable powder distributed through the mass.

## IODINE TRICHLORIDE

Brenken, B. 8, 487.

Connect a large-necked flask or double-necked receiver with both a chlorine generator and a small retort holding 15 grammes iodine, taking care to leave an outlet for the air. Fill the receiver with dry chlorine, and slowly distil the iodine, generating chlorine in the meanwhile as fast as it is used up so as to always have an excess of chlorine in the receiver. When all the iodine has been used up, drive out the excess of chlorine by passing air through the apparatus. Remove the crystals from the walls, and keep in sealed tube. The portion which cannot be loosened may be dissolved in water and the solution kept.

## IODIC ACID

Kämmerer, J. pr. 79, 94.

Heat gently in a hard glass flask 15 grammes iodine and 80 grammes fuming nitric acid, passing a current of air through the liquid. If the iodine does not all dissolve, add more fuming nitric acid from time to time until it does. Evaporate to a small bulk, and recrystallize from rather dilute hot nitric acid until the product is white.

## POTASSIUM FLUORTITANATE

Wöhler, A. ch. p. (3) 29, 185.

Heat to redness 25 grammes rutile, and throw into cold water. Dry, reduce to a fine powder, mix with 50 grammes potassium carbonate and fuse in a platinum crucible placed inside of a Hessian crucible. Cool, powder, and dissolve in hot dilute

hydrofluoric acid, avoiding much excess of the latter. Evaporate to small bulk and allow to cool. Decant, wash the potassium fluortitanate with ice-cold water, and purify by recrystallization.

### TITANIC OXIDE

Wöhler, A. ch. p. (3) 29, 186.

To the cold mother liquor obtained in preparing potassium fluortitanate add ammonia in slight excess, and filter at once from the brown precipitate. Heat to boiling, collect and dry the white precipitate.

### SULPHUR MONOBROMIDE

Muir, Soc. 28, 845.

Wash and dry carefully 20 grammes flowers of sulphur, and add it gradually to 50 grammes bromine, shaking thoroughly after each addition. Distil carefully, heating to  $225^{\circ}$ - $230^{\circ}$ . Purify by redistillation.

## NITROGEN IODIDE

Mallet, Ch. N. 39, 257.

Dissolve 2 grammes iodine in 20 grammes warm alcohol, and pour into 200 c.c. cold water. Wash the powder carefully with water, put it into a beaker, pour upon it 50 c.c. strong ammonia water at zero, and decant. Repeat twice, using no rod or other solid to stir. Wash with alcohol two or three times, then with absolute alcohol, and finally with ether. Divide into small portions before the final washing. Care!!! The nitrogen iodide cannot be kept.

## NITROGEN TETROXIDE

Gay-Lussac, A. ch. p. 1, 405.

Powder and dry 100 grammes lead nitrate. Heat it in a hard glass tube or retort to dull redness, passing the fumes into an empty

wash bottle kept at 30°, then into a U-tube containing fused calcium nitrate, and finally into a dry U-tube kept in ice water and protected from the moisture of the air by a calcium nitrate tube. Pass dry air or oxygen into the liquid thus obtained until it is straw colored. Keep in sealed tubes. Care must be taken to protect the nitrous fumes from organic matter; therefore corks must be wrapped in asbestos paper. Where glass tubes are connected by rubber, wrap the glass in asbestos paper and put the rubber over it, tying with string if necessary.

## NITRIC ANHYDRIDE

Weber, J. pr. (2) 6, 342.

Berthelot, Bl. (2) 21, 53.

To 200 c.c. concentrated colorless nitric acid add slowly 100 c.c. sulphuric acid, keeping cold. Distil gently until 150 c.c. passes

over. To the distillate add 100 c.c. sulphuric acid, and again distil, collecting 100 c.c., to which add 75 c.c. sulphuric acid, and again distil, collecting 50 c.c., which must be carefully protected from moisture and organic matter. Put the distillate into a tubulated retort, and add 30 grammes phosphoric anhydride mixed with beads, keeping below zero. When the whole is thoroughly mixed keep at zero for a few minutes, then allow it to come gradually to the temperature of the room, and finally heat in a water-bath until nitric anhydride comes over. Collect in a small flask kept in ice, and use at once. If the distillation is carried too far a liquid collects on the crystals of nitric anhydride. It should be poured off. It is not as a rule necessary to make connection between the retort and receiver tight to exclude moisture. Organic matter must be rigidly excluded and a vent

provided for the escape of gas. Usually nitric anhydride comes over when the temperature of the water-bath reaches 60°. It must not get above 70°, and should be kept as near 60° as is consistent with the distillation of nitrogen pentoxide.

## **SULPHURIC ANHYDRIDE**

Osann, D. 151, 158.

Weber, P. A. 159, 315.

Into a retort about one-third full of fuming sulphuric acid put a platinum spiral long enough to project above the surface of the liquid and distil, collecting the first fourth. Redistil and keep the product\* in sealed tubes. While distilling protect\* from moisture and organic matter. Leave a vent for the escape of gas. As a precaution put a porcelain jar under the retort.

## ALUMINIUM CHLORIDE

Escales, B. 30, 1314.

Fit a tubulated bell-jar with a glass plate, and cover the glass plate within the jar with asbestos paper. Put upon the asbestos paper an evaporating dish containing 30 grammes aluminium filings. Pass in hydrochloric acid, and as soon as all the air is expelled heat a little aluminium to redness, and lifting the stopper drop it upon the aluminium in the dish. The action continues without further heating if a lively current of hydrochloric acid is passed in. Should the action stop, pass in hydrochloric acid until all the hydrogen is out, and drop in more aluminium heated to redness. When all the aluminium is used up remove the aluminium chloride from the sides and bottom, and keep in tightly closed bottles.

## POTASSIUM PERMANGANATE

Aschoff, J. pr. 81, 29.

Böttger, J. pr. 90, 157.

Graeger, J. pr. 96, 169.

In an iron dish fuse 20 grammes potassium hydroxide, 5 grammes potassium chlorate, and 5-6 c.c. water. Remove the flame, and slowly add 20 grammes powdered manganese dioxide, stirring constantly. Heat to dull redness until the mass is hard and dry. Repeat three or four times with new portions, unite, and powder. Boil the powder with 2 litres water, leading carbon dioxide and air into the boiling solution until a drop leaves a red mark on paper. Filter through glass wool or a Pasteur-Chamberland filter, wash the residue thoroughly, and evaporate until crystals form on cooling. Allow to stand twenty-four hours, pour from the crystals, concentrate the mother liquor, and

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again allow to crystallize, repeating until the crystals are plainly not permanganate. Redissolve all the crystals in boiling water, filter through a Pasteur-Chamberland filter, and recrystallize, excluding all organic matter. Or redissolve all the crystals in cold water, let stand a week, decant carefully from the mud, filter through glass wool, and evaporate to crystallization, excluding organic matter.

### **CRYSTALLIZED SILICON**

Caron, A. ch. p. (3) 63, 26.

Deville & Caron, A. ch. p. (3) 67, 437.

Vigouroux, A. ch. p. (7) 12, 50.

Mix 75 grammes potassium fluorsilicate, 100 grammes granulated zinc, and 20 grammes sodium in small pieces. Throw the mixture into a red-hot crucible, and cover immediately. Unless the contents of

the crucible become liquid at once, heat to fusion and stir with a pipe-stem for a minute or two. Cool slowly, separate the lump of zinc, wash it carefully, and treat with commercial hydrochloric acid. Wash the residue, heat with concentrated nitric acid for a few minutes, wash thoroughly, heat with hydrofluoric acid, wash and dry.

## SILICOCHLOROFORM

Friedel & Ladenburg, A. 143, 118.

Gattermann, B. 22, 186, 190.

Gattermann & Weinlig, B. 27, 1943.

Vigouroux, A. ch. p. (7) 12, 31.

Dry 25 grammes magnesium in the air-bath at 150°, and while still warm (60° to 70°) mix with 40 grammes warm sand previously heated to redness, and then placed in a desiccator. Put the mixture into a small, flat, iron dish (sand-bath), pour upon it a

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coating of dry magnesium oxide, and cover with an iron plate or asbestos board. Heat to redness in one place with a Bunsen burner. As soon as action begins the part heated glows, and the burner may be removed as the action continues throughout the whole mass. When cold throw into cold water, and add hydrochloric acid carefully (Hood). Decant, add concentrated hydrochloric acid, and boil for half an hour. Cool, decant, and wash with hot water until free from magnesia and hydrochloric acid. Put the impure silicon into a combustion tube, and heat to redness in a current of hydrogen. Cool to dull redness, connect one end with a receiver (U-tube or spiral) and the other with a generator of gaseous hydrochloric acid. Put the receiver into ice and brine. To its free end attach a drying tube, and to this a delivery tube dipping into water. Pass a steady stream of

the carefully dried hydrochloric acid, keeping the combustion tube at or just below dull red heat. The water is soon covered with flaky silico-formic anhydride, and unless the delivery tube is dry and wide it soon gets stopped up. A mixture of chlorides of silicon collects in the receiver. If the temperature within the combustion tube was not too high, two-thirds of this is silico-chloroform. It may be obtained pure by redistillation. The distilling flask must not be heated with the naked flame, but placed in a water-bath kept at  $40^{\circ}$ - $50^{\circ}$ . Boiling point of silicochloroform,  $37^{\circ}$ - $38^{\circ}$ .

## **HYDROGEN PERSULPHIDE**

Sabatier, Bl. (2) 44, 169.

Rebs, A. 246, 356.

Dissolve 40 grammes potassium hydrate in 125 c.c. water, saturate with hydrogen

sulphide, and boil in a flask with sulphur as long as any dissolves. Allow to stand until clear, and pour the clear solution slowly into an ice-cold mixture of 60 c.c. each hydrochloric acid (S. G. 1.18) and water. Allow to stand in a separating funnel surrounded by ice until the oil settles. Collect the oil, wash it with ice water, and dry with filter paper. Distil carefully at a pressure of 40 to 80 mm. Boiling-point below 80°.

## BARIUM OXIDE

Fourcroy & Vauquelin, A. ch. 21, 277.

Berthier, A. ch. p. 22, 231.

Put 2 kilos heavyspar and 500 grammes charcoal, carefully mixed, into a Hessian crucible, cover with a layer of powdered charcoal, and heat to bright redness for two hours. When cool throw into cold water and add slowly dilute nitric acid, avoiding

much excess of acid (Hood). When all is in, heat to boiling, filter while hot, and evaporate to small bulk. Purify the barium nitrate by crystallization, and bring it little by little into a hot porcelain or graphite crucible coated with barium sulphate. Heat to quiet fusion, put the crucible into a tight iron box to cool, separate the core from the outer layer, break it up and keep in tightly stoppered bottles.

## **HYDRATED BARIUM PEROXIDE**

Thénard, A. ch. p. 8, 306.

Boussingault, C. r. 32, 261.

Thomsen, B. 7, 73.

Heat barium oxide in a combustion tube to dull redness, and pass in a rapid current of moderately dry oxygen, free from carbon dioxide, as long as any is absorbed. Cool and dissolve in the calculated quantity of

ice-cold, dilute hydrochloric acid. Add solution of barium hydroxide little by little until the precipitate formed is perfectly white. Then filter, and to the filtrate add baryta water to complete precipitation, avoiding, however, any excess of barium hydroxide. Wash the precipitate with ice water, and dry on clay plates.

## HYDROGEN DIOXIDE

Thomsen, B. 7, 73.

Wolfenstein, B. 27, 3307.

Spring, Z. Anorg. 8, 424.

Brühl, B. 28, 2853.

Cool a mixture of 50 c.c. sulphuric acid and 500 c.c. water with ice and salt, and add slowly hydrated barium peroxide until almost neutral. Filter and add more peroxide until barium chloride no longer gives a precipitate. Test for barium salts with sul-

phuric acid, and if present add sulphuric acid. By thus alternating if necessary a solution may be obtained giving no test for either barium or sulphuric acid. Allow to stand in the refrigerator for a day, filter and evaporate on the water-bath in a smooth platinum or porcelain dish until the liquid begins to decompose, then cool rapidly, and concentrate further by distillation in vacuo.

## HYDROXYLAMINE SULPHATE

Divers, Soc. 69, 1665.

Dissolve 75 grammes commercial sodium nitrite and 140 grammes crystallized sodium carbonate in as little ice water as possible, and lead in sulphur dioxide until just acid, agitating thoroughly, and keeping the temperature  $2^{\circ}$ – $3^{\circ}$  below zero by immersing in brine and ice. Add a few drops of sulphuric acid, and heat very slowly to between  $90^{\circ}$

## 50 *INORGANIC CHEMICAL PREPARATIONS*

and 95°. Keep at 90°–95° for about two days, or until all the sodium oxamido-sulphonate has been hydrolyzed. (To determine this add to some of the solution an excess of barium chloride, filter, add to the filtrate some potassium chlorate, and boil for a few minutes; if no precipitate forms the action is over.) When the hydrolysis is completed neutralize with sodium carbonate, using methyl orange as indicator; evaporate until the solution weighs 800–825 grammes, and cool to 5°–8° below zero. Separate from the sodium sulphate, and evaporate until the solution yields at the ordinary temperature abundant crystals of hydroxylamine sulphate. Powder the crystals of sodium sulphate which first separated, wash with little ice water, and add washings to the mother liquor from the hydroxylamine sulphate. Work this up carefully. Purify the crude hydroxylamine sulphate by recrystallization. Devise

a method for getting hydroxylamine chloride from the sulphate, and try it.

## PHOSPHONIUM IODIDE

Baeyer, A. 155, 269.

Dry carefully 100 grammes phosphorus in a 500 c.c. tubulated retort, add sufficient carbon disulphide to dissolve it, and then slowly and carefully 170 grammes dry iodine, keeping the whole cold, and the apparatus filled with carbon dioxide. Distil the carbon disulphide very slowly in a water-bath until it is entirely driven off. Allow to cool, and by means of a long, thin-walled glass tube connect the retort with a receiver protected by a drying tube, which communicates with the outer air or a good draught. Bring a drop funnel into the tubular, and drop in very slowly 60 grammes water. Heat until no more phosphonium iodide

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comes over, or until it no longer comes over white. Transfer the phosphonium iodide, most of which will be found in the glass tube, as quickly as possible to tightly stoppered bottles. The apparatus must be constantly full of carbon dioxide.

Make three or four of the following substances, consulting the original articles for methods of preparation:—

Allotropic Forms of Silver. M. Carey Lea, Am. J. Sc. (3) **37**, 476.

Ammonium Peroxide. Melikoff and Pisarjewsky, B. **30**, 3144; Z. Anorg. **18**, 89.

Calcium. Moissan, C. r. **126**, 1753.

Chromium. Moissan, C. r. **119**, 185.

Ferrotitanium. Goldschmidt, A. **301**, 19.

Hydrazoic Acid. W. Wislicenus, B. **25**, 2085.

Hydroxylamine. Lobrey de Bruijn, Rec. **II**, 18.

Nickel Carbonyl. Mond, Langer & Quincke, Soc. **57**, 750.

Potassium Persulphate. Marshall, Soc. **59**, 772.

Uranium. Moissan, A. ch. p. (7) **9**, 268.

## TABLE OF SOLUBILITIES

When the aqueous solution is saturated at the temperature indicated it contains for every 100 grammes water (X) grammes salt (calculated in every case as anhydrous salt).

	TEMPERATURES INDICATED.		
	Zero.	15°.	100°.
Ammonium Carbonate . . . . .	—	100	—
Ammonium Chloride . . . . .	29	35	77
Ammonium Hydrogen Carbonate	12	18	—
Ammonium Nitrate . . . . .	97	161	—
Ammonium Sulphate . . . . .	71	75	97.5
Barium Chloride . . . . .	31	34.5	59
Barium Dithionate . . . . .	10	20	90
Barium Hydrate . . . . .	1.6	3.1	50
Boric Acid . . . . .	—	3.3	31
Calcium Chloride . . . . .	50	66	155
Calcium Hydrate . . . . .	0.17	0.16	0.08
Calcium Sulphate . . . . .	0.19	0.2	0.17
Chrome Alum . . . . .	—	8 to 9	—
Copper Sulphate . . . . .	15	20	73
Cupric Chloride . . . . .	—	75	—
Cuprous Chloride . . . . .	—	almost insol.	—
Iodic Acid . . . . .	—	very sol.	—
Lead Nitrate . . . . .	36.5	48	127
Manganous Chloride . . . . .	—	67	123
Manganous Dithionate . . . . .	—	sol.	—
Manganous Sulphate . . . . .	55	65	53
Mercuric Chloride . . . . .	5	7	54
Potassium Bichromate . . . . .	5	10	100

	TEMPERATURES INDICATED.		
	Zero.	15°.	100°.
Potassium Carbonate . . . . .	90	110	156
Potassium Chlorate . . . . .	3.3	6	56
Potassium Chloride . . . . .	28	33	56
Potassium Fluortitanate . . . . .	0.5	1	—
Potassium Hydrogen Sulphate . .	36	46	121
Potassium Nitrate . . . . .	13	26	247
Potassium Nitrite . . . . .	—	very sol.	—
Potassium Permanganate . . . . .	—	6	—
Potassium Sulphate . . . . .	8.5	10.3	26
Silver Hyponitrite . . . . .	—	almost insol.	—
Silver Nitrate . . . . .	120	200	1000
Sodium Carbonate . . . . .	7	16.5	45.5
Sodium Chloride . . . . .	35.6	35.8	39.8
Sodium Hydrogen Carbonate . .	7	9	—
Sodium Hydrogen Phosphate . .	2.5	6	99
Sodium Nitrate . . . . .	73	85	180
Sodium Nitrite . . . . .	—	very sol.	—
Sodium Sulphate . . . . .	5	13	42
Sodium Sulphite . . . . .	14	20	—
Sodium Tetraborate . . . . .	1.5	4	55
Sodium Thiosulphate . . . . .	47	65	—
Strontium Chloride . . . . .	44	51	102
Strontium Hydrate . . . . .	0.4	0.66	21.8

The figures given have been largely taken from Comey's *Dictionary of Chemical Solubilities*. In most cases fractions have been omitted. Where two or more observers give different data, an average has generally been struck.



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